THERMOELECTRIC STUDIES ON COBALT ANTIMONIDE BASED COMPOSITE

Final Synopsis

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By

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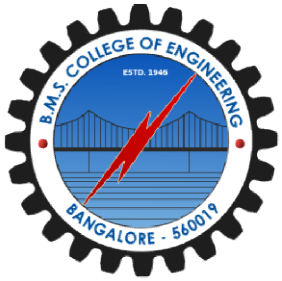

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I. Introduction

Thermoelectric (TE) technology plays an important role in the fields of power generation and solid-state cooling due to the inter-conversion of thermal energy into electrical energy and vice-versa. Among various existing clean energy conversion technologies, thermoelectric material-based devices offer several advantages such as high-power density, high reliability, no moving parts and zero noise. The performance of TE materials at a particular temperature T, is evaluated by the dimensionless figure of merit, $ZT=S^2T/\rho\kappa$, where S is the Seebeck coefficient in V/K, ρ is the electrical resistivity in Ω.m, T is the temperature in Kelvin and κ is the total thermal conductivity in W $m^{-1}K^{-1}$. The total thermal conductivity can be written as, $\kappa = \kappa_e + \kappa_l$, where κ_e and κ_l are the electronic and lattice contributions, respectively. However, all these parameters are closely related to the electronic and crystal structure of a material and they play a crucial role in achieving a large ZT value. Therefore, to obtain better ZT value, it is preferable to achieve high Seebeck coefficient, low electrical resistivity and thermal conductivity [1-3].

The waste heat energy harvesting achieved by employing Thermoelectric Generator (TEG) is considered as a potential energy source for small scale applications in near future. The first generation of TEGs employed were semiconducting thermoelectric like Bi_2Te_3, PbTe, GeSn, GeTe, $CoSb_3$ and SiGe which are efficiently used in deep-space missions and also in photonic and optoelectronic applications [4-11]. However, due to low efficiency of TEG compared to conventional power generators, micro-thermoelectric generators have been developed using complementary metal oxide semiconductor (CMOS) technology [9-11]. Moreover, Bi_2Te_3 material is not suitable for industrial applications at temperatures beyond 250 °C, while SiGe has low conversion efficiency between 200-700 °C temperature regimes and PbTe substance is toxic to the human body and may cause environmental pollution [4-11]. Hence, a search for potential thermoelectric materials for TEG application is still underway. In recent times, TE performance of nanostructured materials like silicides, metal oxides, intermetallics, chalcogenides, clathrates, half-Heuslers and skutterudites have been studied for possible application in power generation [12-17]. Among the various skutterudite TE materials investigated, $CoSb_3$-based materials have garnered attention due to its good thermoelectric characteristics, intermediate temperature operation (500-900K), cost effectiveness, natural abundance, low environmental impact, long-term stability, mechanical and chemical resistance to high temperatures, and versatility in doping

and non-toxic nature. These interesting characteristics invoke $CoSb_3$ as a potential candidate for power generation applications.

Typically, skutterudite $CoSb_3$ has the general structural formula of MX_3, where M stands for the transition atom (Co, Rh, Ir) and X represents the pnicogen atom (P, As, Sb) and presents space group $Im\overline{3}$. The crystal structure possesses two intrinsic voids per unit cell, which can be filled with alkaline earth or rare earth elements to form filled-skutterudites. $CoSb_3$ is a narrow band gap semiconductors material with a bandgap energy of about 0.22 eV. It also depicts large carrier concentration, high mobility, electrical conductivity, large carrier effective mass and Seebeck coefficient owing to its flat energy band [17-19]. However, thermal conductivity of $CoSb_3$ is relatively high (~10 W $m^{-1}K^{-1}$ at 300 K) which results in low ZT value and hence affects thermoelectric performance [20]. Over the decades, many researchers have reported the different approaches to decrease the thermal conductivity by doping at the Co or Sb site [21, 22], filling the guest atoms into the voids [23, 24], inclusion-dispersion in $CoSb_3$ [25-27] and nanostructuring [28, 29]. However, reducing lattice thermal conductivity alone by enhancing phonon scattering without substantial influence on Seebeck coefficient and electrical conductivity is the best way to achieve low thermal conductivity which in turn leads to better ZT factor. Skutterudite structure allows broad tuning of electronic and lattice properties. The skutterudite open structure provides two large voids in the unit cell that can be filled/multi-filled by foreign species such as alkaline-earth and rare-earth elements. These filler atoms remain loosely bound and exhibit "rattling" motion inside the cage structure, hence leads to enhancement of phonon-scattering which thereby accounts for the reduction in thermal conductivity [17, 23, 24]. In most of the research work reported in recent times, nanostructuring has been depicted as potential method to achieve low lattice thermal conductivity by increasing grain boundaries [30]. Nanosized $CoSb_3$-based skutterudites are commonly prepared by several synthesis methods that include ball milling [31, 32], sol-gel [33], co-precipitation [34], hydrothermal method, solvothermal method [35-38] and so on. For the past two decades, researchers have focused on creating new processing technologies in order to provide more effective and large-scale applications for the mass market [39]. In this regard, fine-grained polycrystalline materials were prepared using traditional powder metallurgy and chemical alloying methods [40]. However, these approaches are convoluted and time-consuming. Therefore, the present study aims at evaluating the feasibility of the preparation method and investigating the morphology, structure

and TE properties of $CoSb_3$ prepared by ball milling and solvo-hydrothermal methods, respectively.

II. Motivation of the proposed research work

The $CoSb_3$ based skutterudite has been considered a promising high-efficiency TE material for mid-temperature (500-800 K) applications because of its high Seebeck coefficient, high mobility and narrow band gap. As compared to the other thermoelectric materials, $CoSb_3$ does not contain toxic material which makes it a near-future potential candidate to be used in the fabrication of thermoelectric generator. However, the strong Co-Sb covalent bond results in a high lattice thermal conductivity (~10 W $m^{-1}K^{-1}$ at 300 K) and hence leads to a low thermoelectric figure of merit. So, the motivation for the proposed research work is to increase the thermoelectric efficiency of $CoSb_3$ by minimizing the lattice thermal conductivity to the lowest possible value and increasing the power factor by optimizing carrier concentrations. Multiple approaches are proved to be more beneficial for increasing the efficiency of thermoelectric properties. The $CoSb_3$ skutterudite can be structurally modified by adding impurity atoms to Sb sites on the pnicogen rings which can enhance thermoelectric characteristics. Theoretical calculation of electronic structure demonstrated a consistent shift in the Fermi level towards the conduction band in the presence of dopants (Se and Te), which resulted from the addition of one electron to the system without appreciably altering the electronic structure. Hence, Te doping at Sb site not only changes the conduction mechanism from p-type to n-type, but also improves ZT by reducing resistivity and thermal conductivity. Moreover, inclusion of nanoparticles in the bulk matrix results in a considerable reduction in lattice thermal conductivity due to induced phonon scattering at interfaces. Furthermore, this method can improve the Seebeck coefficient by filtering low-energy charge carriers at the potential barrier created at the interfaces. As a result, the nanocomposite method can control the Seebeck coefficient and decrease the lattice thermal conductivity. The combination of multiple approaches has been shown to be more effective in optimizing thermoelectric characteristics. The proposed research work combines doping and nanocomposite approaches to improve the thermoelectric performance in n-type $CoSb_3$ materials. The findings of these approaches are rigorously evaluated and compared to the structural and transport property analyses.

III. Aim and Objectives of the proposed research work

The main aim of the proposed research is to synthesize thermoelectric skutterudite nanomaterials with high purity, crystallinity and study their transport properties. Further, focus is on the study of efficiency of thermoelectric nanomaterials and its possible application in thermoelectric power generator for green energy technology.

In order to realize the objectives it is proposed:

- To synthesize the pristine and filled $CoSb_3$ skutterudite nanomaterials with optimized conditions by solvo-hydrothermal method
- To investigate in detail the structural, morphological and optical properties of the as-synthesized nanomaterials
- To consolidate the solvo-hydrothermally synthesized nanopowders by microwave sintering with optimized conditions and investigates the transport properties such as electrical resistivity and Seebeck coefficient of the as-synthesized nanomaterials
- To synthesize the nanostructured $CoSb_3$ skutterudite by ball milling method and compare structural, morphological, optical and transport properties (electrical resistivity, Seebeck coefficient and thermal conductivity) of $CoSb_3$ skutterudite nanomaterials prepared via a solvo-hydrothermal method
- To synthesize doped and nanocomposites of $CoSb_3$ matrix by ball milling method followed by spark plasma sintering technique, and study their thermoelectric performance

IV. A brief overview of the proposed thesis chapters

Chapter I: Introduction

Chapter I presents a broad outline of the introduction, basic principles, significance, and applications in the field of thermoelectric technology. Also, this chapter provides a brief overview of the background of skutterudites-based thermoelectric materials and their applications in thermoelectric devices. A comprehensive review of the literature on $CoSb_3$ skutterudite, as well as the advantages of thermoelectric material over traditional materials is provided. Methods for improving thermoelectric characteristics, such as increasing electrical conductivity while decreasing thermal conductivity, are described. The challenges in increasing the ZT value in skutterudite materials by adopting nanostructuring, doping, and composite approaches are discussed in detail.

Chapter II: Experimental procedure and characterization techniques

Chapter II describes the experimental procedure for the synthesis of skutterudite nanomaterials by solvo-hydrothermal method and ball milling technique. The first section of this chapter discusses about structural and morphological characterizations techniques such as powder X-ray diffraction (pXRD), field emission scanning electron microscope (FESEM), and Transmission electron microscopy (TEM). The second section deals with optical and thermal characterizations techniques like Fourier Transform Infrared Spectroscopy (FTIR), UV-VIS absorption spectroscopy, Raman spectroscopy, Thermogravimetric Analysis (TGA), and Differential Scanning Calorimetry (DSC) analysis. The temperature-dependent electrical resistivity, Seebeck coefficient, and thermal conductivity, and also mechanical properties measurement techniques are discussed in the last section.

Chapter III: Structural and optical studies on pristine and Strontium-filled $CoSb_3$ nanoparticles synthesized via solvo-hydrothermal method

In this chapter, pure $CoSb_3$ and strontium-filled $CoSb_3$ nanoparticles (Sr_yCoSb_3, y = 0, 0.025, 0.05, 0.075 and 0.1) synthesized by a solvo-hydrothermal method are discussed. Powder x-ray diffraction (pXRD) analysis reveals a cubic phase of $CoSb_3$ with space group $Im\overline{3}$. The Sr-filled

samples show a slight peak shift and broadening of the high-intensity peak at 31.249º corresponding to the (013) plane, which can be attributed to the interaction of Sr atoms filled into voids of the $CoSb_3$ cage-like structure with some of the lattice vibrations in the structure. Field emission scanning electron microscopy (FESEM) images show as-synthesized nanoparticles in the range of 50–160 nm, and energy-dispersive x-ray spectroscopy (EDX) analysis reveals the chemical composition of Sr-filled $CoSb_3$. Fourier transform infrared spectroscopy (FTIR) studies confirm vibrational modes below 1000 cm^{-1} corresponding to Co-Sb and cobalt complexes in both filled and unfilled $CoSb_3$ nanoparticles. UV–Vis absorption analysis indicates a peak shift towards the longer-wavelength region (red shift) and a decrease in the optical band gap as a function of an increase in Sr filling concentration. This can be considered as strong evidence for successful filling of voids in the cage-like structure of $CoSb_3$ by strontium.

Chapter IV: Substantial enhancement of thermoelectric power factor of undoped $CoSb_3$ sktterudites processed by microwave sintering

In this chapter, a new strategy of combining the solvo-hydrothermal method with microwave sintering (MWS) technique were successfully employed to synthesize single phase p-type $CoSb_3$ skutterudite. The microwave sintered bulk material with uniform grain size of 190 nm to 230 nm, and a compaction density of >80% were obtained. The effect of microwave sintering conditions on the thermoelectric properties is discussed in detail. All the samples show the enhanced power factor. The disordered structure was introduced into the ordered $CoSb_3$ nanocrystalline by varying the microwave sintering temperature and time duration in order to improve the Seebeck coefficient meanwhile maintaining excellent electrical conductivity. The highest power factor of 1508.6 $\mu Wm^{-1}K^{-2}$ was achieved at 663 K for the microwave sintered sample. The microwave-sintered samples show an enhanced thermoelectric power factor compared to conventional sintering techniques. This technique offers an effective strategy for the large-scale production of skutterudite materials at a low cost.

Chapter V: Influence of synthesis method and processing on the thermoelectric properties of $CoSb_3$ skutterudites

In the present work, single phase $CoSb_3$ skutterudite nanomaterials were prepared via a ball milling method, and their thermoelectric characteristics were compared with the samples

synthesized by solvo-hydrothermal method. Thermoelectric transport properties were recorded in the temperature regime of 300-830 K. Both samples exhibit p-type conduction behavior with a positive sign of Seebeck coefficient. $CoSb_3$ ball milled sample exhibit higher resistivity of 127×10^{-5} Ω.m at 300 K as compared to $CoSb_3$ solvo-hydrothermal sample with 4.85×10^{-5} Ω.m. However, $CoSb_3$ ball milled sample shows enhanced Seebeck coefficient of 183 μV/K at 473 K, whereas $CoSb_3$ solvo-hydrothermal sample depict 98 μV/K at 650 K. Furthermore, the total thermal conductivity values of sintered $CoSb_3$ ball milled and $CoSb_3$ solvo-hydrothermal samples were found to be 3.02 W $m^{-1}K^{-1}$ and 3.23 W $m^{-1}K^{-1}$ at room temperature and reaches a significantly lower value of 2.47 W $m^{-1}K^{-1}$ and 2.46 W $m^{-1}K^{-1}$ at 580 and 670 K, respectively. The dimensionless figure of merit ZT values of ball milled $CoSb_3$ and solvo-hydrothermally obtained $CoSb_3$ are 0.053 and 0.060 at 650 and 700 K, respectively. A systematic tuning of processing parameters by ball milling method can lead to $CoSb_3$ materials with better thermoelectric efficiency.

Chapter VI: Possible reduction of lattice thermal conductivity in n-type $CoSb_{2.875}Te_{0.125}$ skutterudite originating from collaborative adjustment between Indium micro and α-WC nano inclusions

In this chapter, single-phase Te-doped $CoSb_3$ polycrystalline bulk (Indium metal powder) and nanocomposites (α-WC powder) were synthesized via ball milling followed by spark plasma sintering technique. The thermoelectric and mechanical properties of as-synthesized composites were studied. The electrical resistivity reduced from 1200 μ Ω.m for pristine $CoSb_3$ to 12.30, 12.36, 11.82, 12.82, 12.43 and 12.44 μ Ω.m for $CoSb_{2.875}Te_{0.125}$+x (x = 0.33% In, 1% In, 2% In, 4% In and 1% In + 0.33% WC), respectively. At temperature 300K, composite with x = 1% In exhibit the lowest resistivity of 11.82 μ Ω.m. At room temperature, Seebeck coefficient values are -131.7, -129.8, -133.6, -130.7, -126.2 and -132.1 μV/K for $CoSb_{2.875}Te_{0.125}$+x (x=0%, 0.33 % In, 1% In, 2% In, 4% In and 1% In + 0.33% α-WC), respectively. The maximum power factor of 2566 $\mu Wm^{-1}K^{-2}$ and 2482 $\mu Wm^{-1}K^{-2}$ are observed for 1% In and 1% In + 0.33% α-WC composites at 673 K, respectively. Notably, the power factor of 1% In and 1% In + 0.33% α-WC composites is slightly higher (1.05 times) than the $CoSb_{2.875}Te_{0.125}$ sample. The composites with low volume ratios of 1% In and 1% In + 0.33% α-WC have significantly increased power factor, while the lattice thermal conductivity is lowered only for 1% In + 0.33% α-WC composites. The

multi-scale size distribution of grains scatter phonons throughout a wide wavelength range, resulting in extremely low lattice thermal conductivity. As a result, an increased figure of merit of 0.82 was achieved for $CoSb_{2.875}Te_{0.125}$+1% In + 0.33% α-WC composites at 823 K. The results suggest that the doping with composite approach could boost thermoelectric efficiency in n-type $CoSb_3$-based materials.

Chapter VII: Outcome of the proposed research work

This section summarizes the present results and outline of the future work and opportunities in the field of thermoelectric technology. The development of thermoelectric generators from waste heat recovery will have great scientific as well as technological importance.

The outcome of the research work will be presented in national/international conference and data will be published in reputed international journals. A thesis incorporating the results of the above systematic investigation will be submitted to the Mangalore University for the award of Ph.D., Degree.

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List of Conferences/Workshops attended:

1. Participated in QIP-Short Term Course on "Thermoelectric Material and its Applications" organized by CCE, IISc, Bengaluru from 13-19 May 2019.
2. "Effect of Sn doping at Sb sites on the structural and optical properties of Co_2Sb_6 nanostructures" **(Poster presentation) M Uday Kumar**, Swetha R, Murugendrappa M V and Latha Kumari, *International Conference on Advanced Materials (ICAM-2019)*, held at Nirmalgiri College, Kannur, Kerala, during 12-14 June 2019.
3. Participated in TEQIP workshop on "Advanced Materials Characterization Techniques" organized by IIT Hyderabad from 27 Aug -1 Sep 2019.
4. "Synthesis, structure and optical properties of Indium filled $CoSb_3$ nanomaterials" **(Oral presentation) M Uday Kumar**, Swetha R, and Latha Kumari, *International Conference on Multifucntional Materials (ICMM-2019)* held at Geethanjali College of Engineering and Technology, Hyderabad during 19-21 Dec 2019.
5. Participated in "Basic Training Programme in Nano Science and Technology" organized by CeNSE, IISc, Bengaluru from 20-22 Jan 2020.
6. Participated and presented **(Poster presentation)** paper entitled "Structural and Optical properties of Strontium filled $CoSb_3$Skutterudites" **M Uday Kumar**, Swetha R, and Latha Kumari, *International Conference on Materials Genome (ACCMS-ICMG-2020)* organized by SRM University, Andhra Pradesh from 5-7 Feb 2020.
7. Participated and presented **(Poster presentation)** paper entitled "Impact of ball milling duration, Phase transformation and processing on the thermoelectric performance of Cu_2Se" **M Uday Kumar**, Swetha R, and Latha Kumari, *International Conference on Materials Genome-III (ICMG-III)* organized by SRM University, Andhra Pradesh from 22-24 Feb 2024.

List of Journal Publications and Conference Proceedings:

1. "Effect of Sn doping at Sb sites on the structural and optical properties of $CoSb_3$ nanostructures", **M Uday Kumar,** R Swetha, M V Murugendrappa and Latha Kumari AIP Conference Proceedings **2162,** 020054 (2019).
   DOI: **https://doi.org/10.1063/1.5130264**

2. "Fabrication, structure and optical characterization of Ce filled $CoSb_3$ nanostructure by solvothermal method", R Swetha, **M Uday Kumar** and Latha Kumari AIP Conference Proceedings **2162,** 020104 (2019). DOI: **https://doi.org/10.1063/1.5130314**

3. "Synthesis, structure and optical properties of Indium filled $CoSb_3$ nanomaterials", **M Uday Kumar,** R Swetha, and Latha Kumari. J. Phys.: Conf. Ser. **1495,** 012006 (2020).
   DOI: **https://doi.org/10.1088/1742-6596/1495/1/012006**

4. "Structural and Optical Studies on Strontium-Filled $CoSb_3$ Nanoparticles via a Solvo-/Hydrothermal Method", **M Uday Kumar,** R Swetha, and Latha Kumari. Journal of Electronic Materials **50,** 1735–1741 (2021).
   DOI: **https://doi.org/10.1007/s11664-020-08629-2**

5. "Synthesis and characterization of polypyrrole-$Ce_{0.05}CoSb_3$ nanocomposites", R Swetha, **M Uday Kumar** and Latha Kumari, Mater. Today: Proc. **46,** 2934-2939 (2021).
   DOI: **https://doi.org/10.1016/j.matpr.2020.11.398**

6. "Effect of Calcination Temperature on the Structural and Optical Properties of $FeSbO_4$ Nanostructures", B. V. Sahana, **M Uday Kumar,** R Swetha, Ramappa S. Kuri, Latha Kumari, Cryst. Res. Technol. 2300174 (2023).
   DOI: **https://doi.org/10.1002/crat.202300174**

7. "A Comparative Study on the Structural and Optical Properties of $NiMoO_4$ Nanomaterials Synthesized by Hydrothermal and Solvothermal Route", Ramappa Sangappa Kuri, B. V. Sahana, **M Uday Kumar,** R. Swetha, Latha Kumari, Cryst. Res. Technol. **59,** 2300178 (2024). DOI: **https://doi.org/10.1002/crat.202300178**

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